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Effect of holding pressure on densification and mechanical properties of C_f/Mg composites

X. Wei¹, L. Qi^{*1}, L. Ju¹, W. Tian¹, X. Hou² and H. Li³

¹School of Mechanical Engineering, Northwestern Polytechnical University, Xi'an 710072, China

²University of Nottingham, University Park, Nottingham NG7 2RD, England

³School of Materials Science and Engineering, Northwestern Polytechnical University, Xi'an 710072, China

*Corresponding author, email qilehua@nwpu.edu.cn

Abstract

Carbon fibre reinforced magnesium alloy matrix composites were fabricated by using liquid-solid extrusion directly following vacuum infiltration technique. The experimental results showed that the microstructures of C_f/Mg composites depended on the holding pressure. The porosity was reduced gradually, and the densification was improved obviously, respectively, with the increase of the holding pressure. The densification, hardness and Ultimate tensile strength of C_f/Mg composites were significantly improved as the holding pressure increased in the range of 0.1–15 MPa. The densification was not obvious, but the UTS of the C_f/Mg composites decreased gradually as the holding pressure increased in the range of 25–45 MPa. The C_f/Mg composites presented a good performance when the holding pressure was about 15 MPa.

Keywords: Holding pressure, Microstructure, Densification, Mechanical properties, C_f/Mg composites

Introduction

Carbon fibres have been widely used in various composites for its excellent properties. Carbon fibre reinforced magnesium alloy matrix composites (C_f/Mg) exhibit high specific strength and specific stiffness, low thermal expansion coefficient, which produces a good application prospect in aerospace and automobile field [1-3]. C_f/Mg composites can be used as a type of lightweight structural materials to replace the traditional aluminium alloy and magnesium alloy, such as satellite antennas, space mounting plates and optical reflector brackets [4-7]. However, the applications of C_f/Mg composites are restrained due to the existence of some microscopic defects, which are caused by inappropriate fabrication parameters. Hence, it is necessary to study the correlation between the performance of the C_f/Mg composites and the fabrication parameters.

Regarding the fabrication of C_f/Mg composites, liquid fabrication method has become a priority due to the economical consideration, therefore the shrinkage of molten magnesium alloy is an important issue [8]. With liquid fabrication method, the preform can be infiltrated by molten magnesium alloy under a certain pressure due to the good liquidity of metal in the molten state. Liquid fabrication method can be categorised into squeeze casting, pressureless infiltration and vacuum pressure infiltration, etc., according to the pressure way [9-11]. In squeeze casting, molten magnesium alloy is exposed to air, so it is easy to introduce entrapped air within the composites [10]. A good wettability between carbon fibres and matrix metal should be required in the pressureless infiltration [11]. Equipment requirements in vacuum pressure infiltration process are more comprehensive and complex compared with other fabrication methods [12]. Liquid–solid extrusion directly following vacuum infiltration (LSEVI) is a special technique that combines the advantages of traditional vacuum infiltration and squeeze casting process, in which magnesium alloy melting, preform

preheating, vacuum infiltration and liquid–solid extrusion can be integrated in an experiment system [13-15]. crystallised and solidified in the carbon fibre preform under an appropriate pressure. However, the introduction of carbon fibres brings a variation in the densification process of C_f/Mg composites [16]. When the infiltration process of molten magnesium alloy in a carbon fibre preform completes, a suitable holding pressure is very critical for promoting the solidification of molten magnesium alloy, reducing the porosity and improving the performance of C_f/Mg composites [17]. In order to optimise the pressure-holding process, it is necessary to evaluate the microstructures and mechanical properties of C_f/Mg composites under different holding pressures. For the pursuit of high quality metal matrix composites, the fabrication methods have been conducted by many scholars. Lee *et al.* [18,19] studied the microstructures of the metal matrix composites (MMC), and the experimental results showed that the fine microstructures gave the possibility of improving the material properties of the MMCs. Seong *et al* [20] carried out the melt infiltration by squeeze casting in processing continuous graphitic fibre reinforced aluminium matrix composites, and a region consisting of coarse dendrites was found in the fibre-free matrix. Sutradhar [21] investigated the solidification characteristics of aluminium alloy matrix composites, and found that the solidification rate was faster in the case of unreinforced matrix alloy. However, the fabrication parameters have to be properly controlled because entrapment and oxidation are more easily occurred in molten magnesium alloy. The excessive pressure can also damage the reinforcement [20]. Hence, it is necessary to study the effect of holding pressure on the performance of C_f/Mg composites.

In this paper, C_f/Mg composites were fabricated by LSEVI technique with different holding pressures. To optimise the holding pressure, the microstructures, densification process and mechanical properties of C_f/Mg composites were investigated.

Materials and methods

Materials

Casting magnesium alloy AZ91D was selected as the matrix material. AZ91D magnesium alloy presented a good liquidity and a wide range of solidification temperature, so it was suitable for the fabrication of C_f/Mg composites. The chemical compositions of AZ91D magnesium alloy were 8.30–9.70% Al, 0.35–1.00% Zn, 0.15–0.50% Mn, ≤0.10%Si, ≤0.03%Cu and balance Mg.

Toray T700-12 K carbon fibres were selected as the reinforcements, which had an average diameter of 7 μm, a density of 1.78 g cm⁻³ and a tensile strength of 3.9 GPa. The effective diameter of carbon fibre bundle is 0.65 mm. The preforms were fabricated by the superposition of T700-12k non-woven fabrics with an arrangement of 2D – 0°/90°, which would be beneficial to maintain structural integrity. The preforms were stitched with carbon fibres bundle along the thickness direction. The volume fraction of the preform was about 40%.

Pyrolytic carbon coating was deposited on the surfaces of carbon fibres to prevent the high temperature oxidation and interface reaction in the fabrication of C_f/Mg composites [22]. The thickness of pyrolytic carbon coating was in the range of 200–300 nm.

Methods

The fabrication of C_f/Mg composites was carried out in a forming mould, which was made of the 3Cr2W8 V steel. The internal diameter of forming mould was 140 mm. The experimental equipment was shown as Fig. 1.

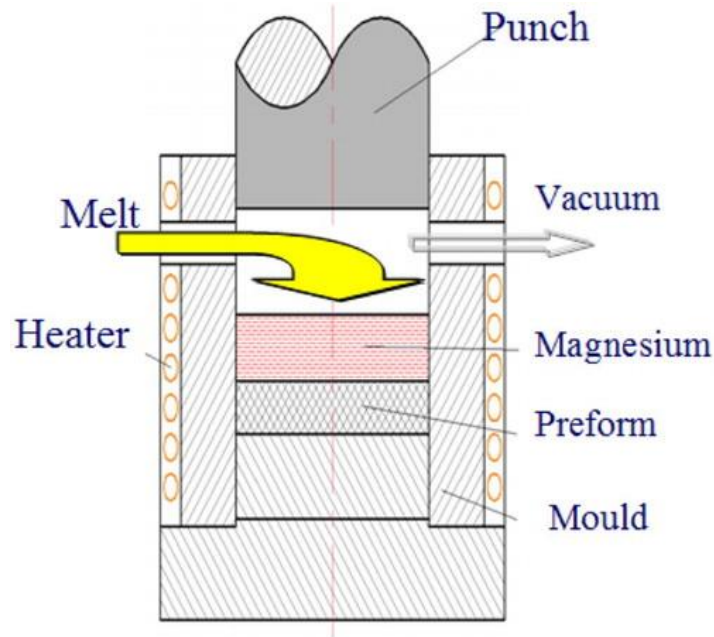


Fig. 1 Schematic diagram of forming mould

The experimental procedures were given as follows: 700–800 g magnesium alloy was melted in a crucible at 720°C, meanwhile the forming mould was preheated to 595–600°C, which ensured a good infiltration of the molten magnesium alloy into the carbon fibre preform. The forming mould was persevered at 595–600°C for 15–25 min, and then vacuumed by a pump. Next, molten magnesium alloy was poured into the forming mould under a gas pressure of 0.15–0.2 MPa [23]. Infiltration pressure was applied to the surface of the molten magnesium alloy, which was produced by a moving punch connected to a hydraulic press machine (as shown in Fig. 1). In this study, the pressure of 20 MPa was applied to the molten magnesium alloy for conducting the infiltration. Holding pressure was conducted for promoting feeding of the molten magnesium alloy after the infiltration. Different holding pressures were applied by the punch until the magnesium alloy solidified totally as the forming mould temperature decreased to 580°C. In the case that the pressure was not applied by punch, the holding pressure was considered as 0.1 MPa. Finally, C_f/Mg composites were removed from the die under room temperature.

Testing

An optical microscope was used to observe the metallographic microstructures of C_f/Mg composites after grinding and polishing. Wolpert 430SVD Vickers hardness tester was used to test the hardness of C_f/Mg composites, and the test load was 196 N. Sartorius-CP225D analytical balance with precision of 1×10^{-4} g was used to test the density based on the Archimedes's principle. UTS tests were conducted on a CMT5304-30KN universal electronic tensile testing machine with the loading rate of 0.5 mm min^{-1} , and three tensile samples were tested in each group. Sample test was based on industry standard HB7617-1998. The dimension of the tensile specimens was $75 \text{ mm} \times 10 \text{ mm} \times 2 \text{ mm}$. TESCAN VEGA II scanning electron microscope was used to observe the microstructures and the fracture surfaces of C_f/Mg composites. The schematic diagram of sampling range was shown in Fig. 2.

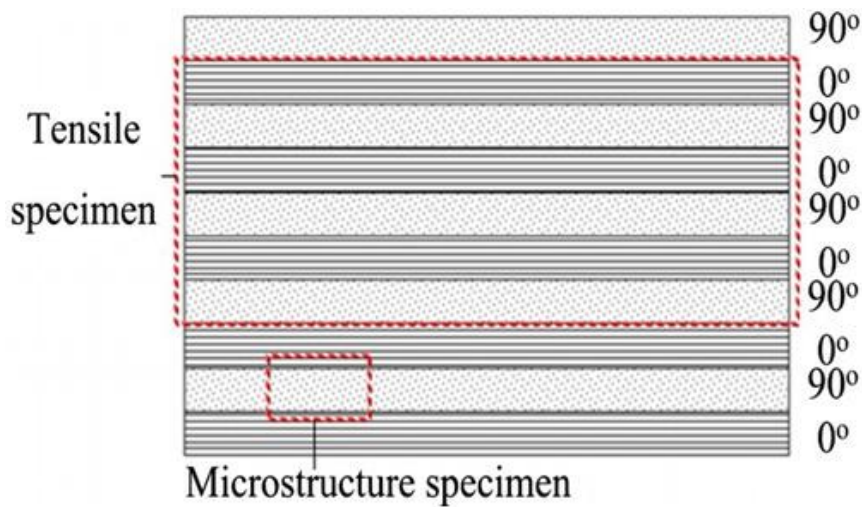


Fig.2 Tensile specimens of C_f/Mg composites

Results and discussion

Effect of holding pressure on the microstructures of C_f/Mg composites

The solidification of the obtained molten C_f/Mg composites should be completed under a certain pressure to avoid shrinkage. Figure 3 illustrated the microstructures of C_f/Mg

composites fabricated under different holding pressures, which showed that the magnesium alloy has been infiltrated into the carbon fibre preform completely.

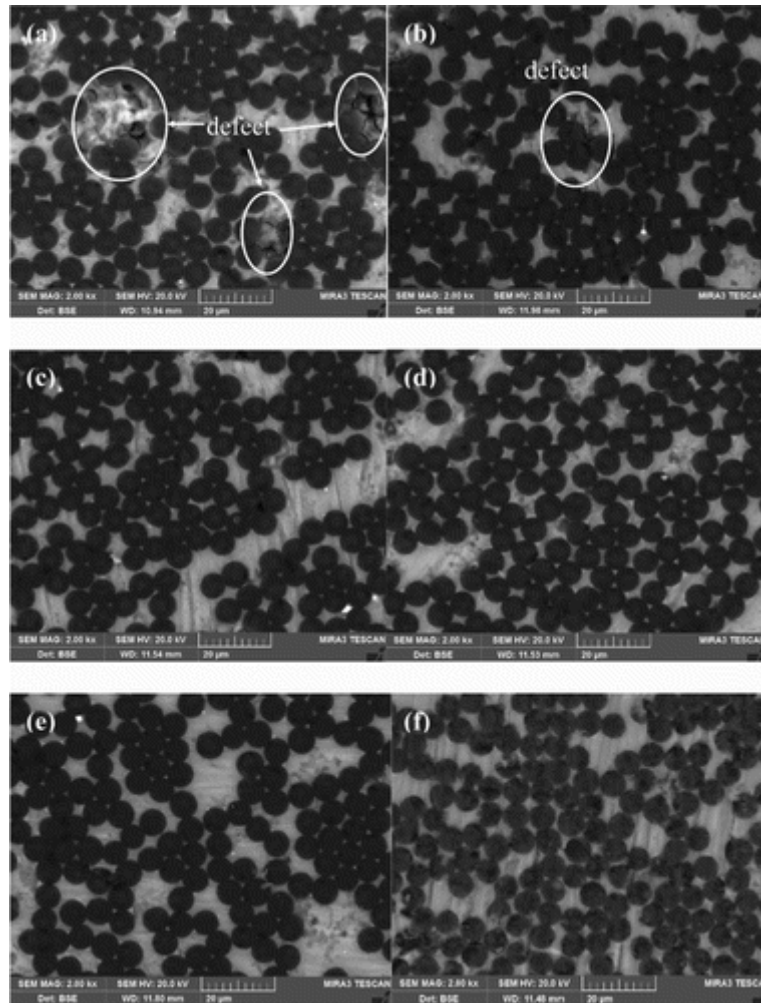


Fig. 3 Microstructures of C_f/Mg composites with different holding pressures: (a) 0.1 MPa, (b) 5 MPa, (c) 15 MPa, (d) 25 MPa, (e) 35 MPa and (f) 45 MPa.

When the holding pressure was 0.1 MPa or low (5 MPa), some shrinkage cracks appeared at the interfaces between the magnesium alloy and carbon fibres, as shown in Fig. 3a and b.

Defects were caused by the uneven shrinkage of molten magnesium alloy around the carbon fibres because of the different thermal expansion behaviours of magnesium alloy and carbon fibres. Linear thermal expansion coefficient of T700 carbon fibre was $7 \times 10^{-6}/\text{K}$, while linear shrinkage rate of magnesium alloy was 1.57% in the solidification process [24]. Then the

shrinkage defects appeared between carbon fibres due to the lack of holding pressure, which led to the micro defects in the C_f/Mg composites. Micro cracks reduced or even disappeared gradually with the increase of holding pressure. When the holding pressure was greater than 15 MPa, the microstructures of C_f/Mg composites were compact, and the microscopic defects and blind angle among carbon fibres have not been found (seen Fig. 3c–f). An appropriate holding pressure could reinforce the bonding between the carbon fibres and magnesium alloy, and prevent the magnesium alloy shrinkage.

The TEM of C_f/Mg composite was shown in Fig. 4, which indicated the interface of carbon fibre/matrix was mainly free of carbides. This was due to the fact that the PyC coating served well as an inhibitor of deleterious carbide formation, even if magnesium alloy containing Al element were used as matrix materials [22, 25].

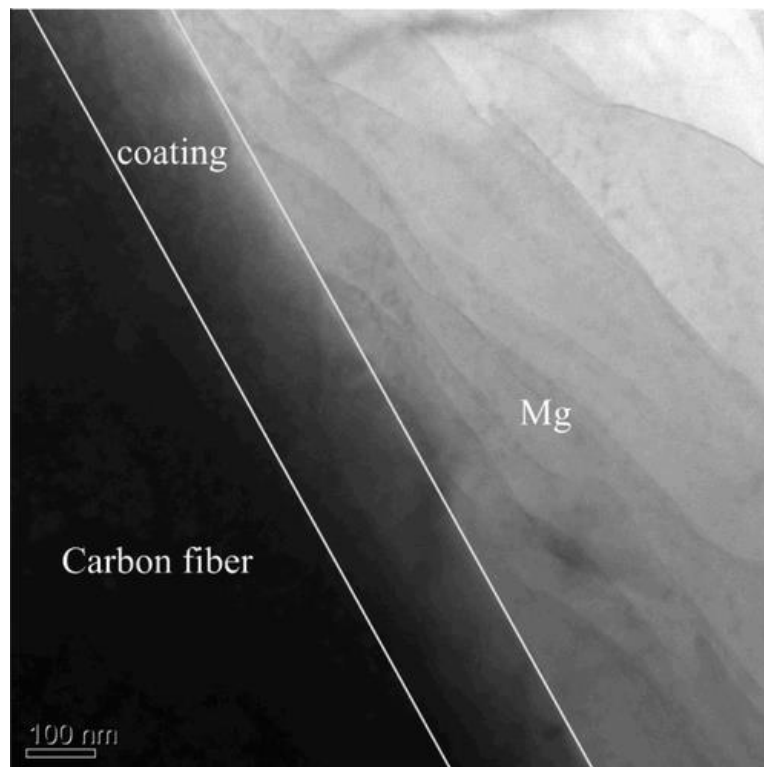


Fig. 4 TEM of C_f/Mg composites

Effect of holding pressure on the densification of C_f/Mg composites

In the densification of the C_f/Mg composites, magnesium alloy was in the semisolid state. The densification of the C_f/Mg composites increased with the growth of holding pressure until 15 MPa, above which the densification was almost constant. The density and hardness of the C_f/Mg composites fabricated with different holding pressures were shown in Fig. 5.

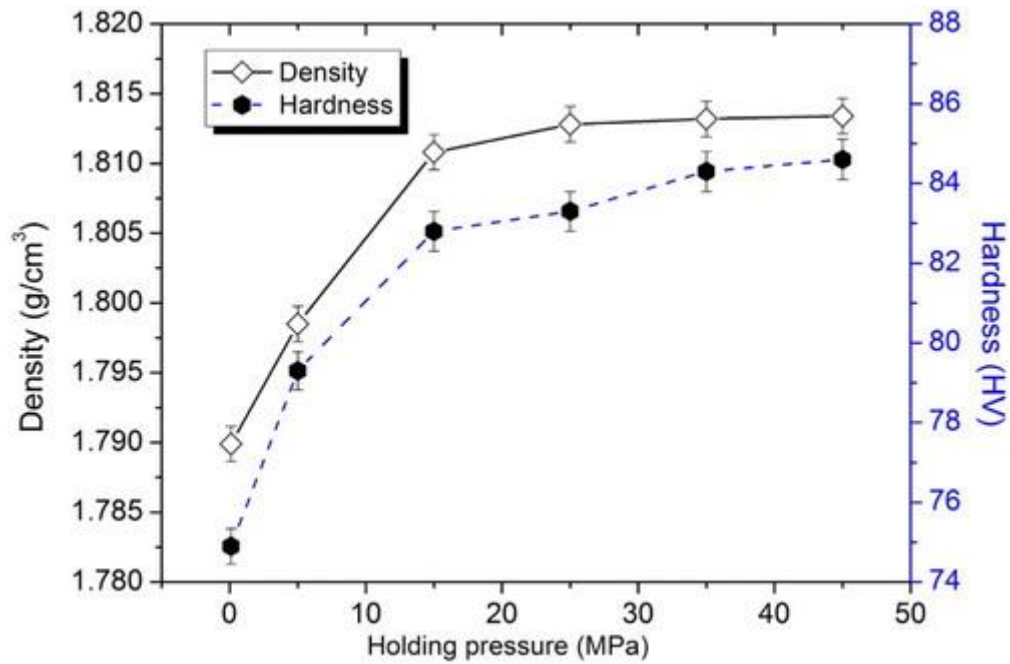


Fig.5 Density and hardness of C_f/Mg composites fabricated under the different holding pressures: *a* densities *b* hardness

When the C_f/Mg composites solidified under the pressure more than 15 MPa, the molten magnesium alloy filled the shrinkage in the dendrites, which helped to eliminate the shrinkage. Holding pressure played a critical role in the densification of C_f/Mg composite. The increase of holding pressure could inhibit the gas entrapment in molten magnesium alloy and reduce porosity, which could make the density and hardness increase [26].

Based on the microstructures shown in Fig. 2, the density and the hardness, further densification of C_f/Mg composites could be less effective when the holding pressure

exceeded 15 MPa. This indicated that 15 MPa was the critical pressure to ensure the complete densification.

Effect of holding pressure on the mechanical properties of the C_f/Mg composites

The UTS of C_f/Mg composites was improved compared with that of casting AZ91D (150 MPa), and the holding pressure had a significant effect on the mechanical properties of C_f/Mg composites. The percentage elongation (2.8–5.4%) of C_f/Mg composites was lower than that of casting AZ91D (8%). Test results were shown in Table 1.

Table 1 Mechanics performance test results

Holding pressure (MPa)	0.1	5	15	25	35	45
UTS (MPa)	251	307	353	335	287	264
Elongation (%)	3.9	3.2	5.4	5.2	3.6	3.8

The mechanical properties and stress–strain curves of C_f/Mg composites with different holding pressure were given in Fig. 6*a* and *b*.

When the holding pressures were 0.1 or 5 MPa, the UTS of C_f/Mg composites were at a low level due to the existence of micro defects, and the stress–strain curve showed a brittle fracture mode. The increase of UTS was significant when the holding pressures were 15 and 25 MPa, which was contributed by the decrease of defects. In the stress–strain curves of the sample fabricated under 15 and 25 MPa, when the stress reached a maximum load, the load curves declined sharply due to the breakage of some carbon fibres. Meanwhile, the remaining carbon fibres began to play a load-bearing role and contributed to a small load increase, then the load fell again resulting from the fracture of other carbon fibres. Thus, a step-like fracture surface was produced with the stress cycle and a slight fluctuation in the stress–strain curve was noticed. This fracture mode indicated that the stress concentration was adjusted by the

interface of C_f/Mg composites [27]. When the holding pressures were greater than 35 MPa, with the increase of holding pressure, the UTS showed a downward trend, and the characteristics of stress–strain curve were consistent with the curve of low holding pressures. In summary, if the holding pressure was greater than 15 MPa, the further holding pressure was not suggested, and it tended to cause ejection force increase and reduce the forming mould life. In order to further determine the influence of holding pressure, the fracture surfaces of C_f/Mg composites were observed, as shown in Fig. 7.

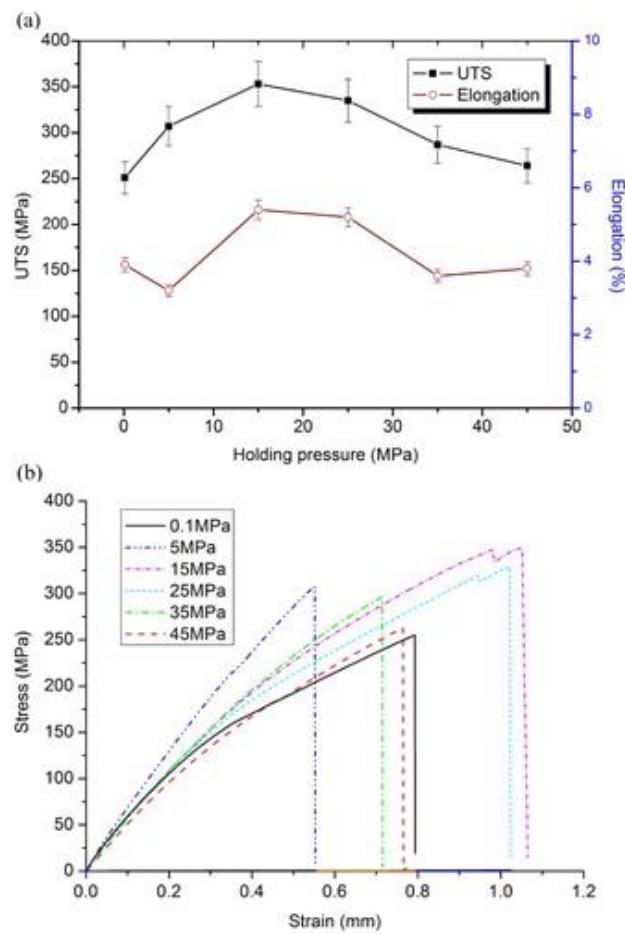


Fig. 6 Mechanical properties and stress–strain curves of C_f/Mg composites solidified with different holding pressures: (a) mechanical properties (b) stress–strain curves

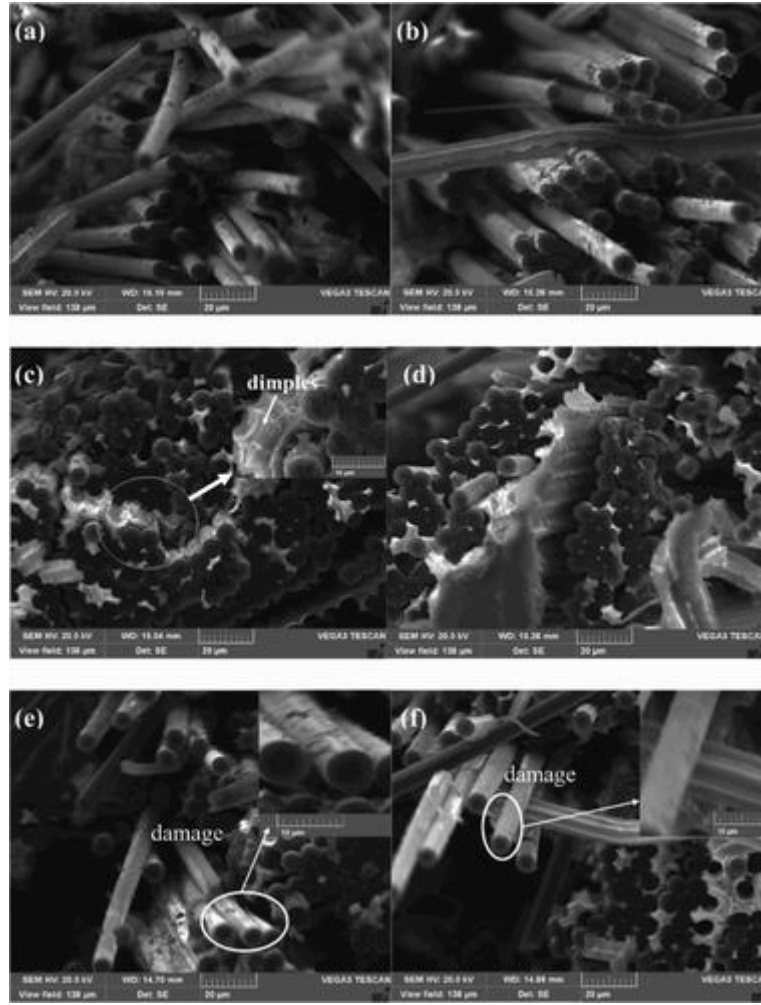


Fig.7 Fracture morphologies of C_f/Mg composites with different holding pressures: (a) 0.1 MPa, (b) 5 MPa, (c) 15 MPa, (d) 25 MPa, (e) 35 MPa and (f) 45 MPa.

The fracture morphologies of C_f/Mg composites were mainly composed of carbon fibre fracture and a small amount of dimples in magnesium alloy [28]. Pull-out of carbon fibres could be observed in Fig. 7a–f. In Fig. 7a and b, many loose carbon fibres could be seen, and the adjacent carbon fibres were not connected by the matrix. The loose fracture surface was induced by the lack of feeding magnesium alloy, which would cause a poor bonding between carbon fibre and magnesium alloy. The dimples of the magnesium alloy and the debonding between carbon fibres and matrix could be found in Fig. 7c and d, which presented an obvious step-like fracture surface. All of these were beneficial to play the full load-carrying role of carbon fibres, which could be seen in the stress–strain curve [29]. The contact angle θ

between carbon fibres and molten magnesium alloy was more than 90° , so the solidification interface would be bulged, and the tip of the dendrite growth tendency was located in the space between adjacent carbon fibres [30]. When the holding pressure was too high, the damage on the carbon fibres occurred, which directly reduced the mechanical properties of C_f/Mg composite materials [31]. In Fig. 7e and f, pyrolytic carbon coating showed partly falling and carbon fibres suffered from obvious damage.

The fracture morphologies analyses also verified that the holding pressure was required to keep in a reasonable value. In order to prevent the damage of carbon fibres in the fabrication of C_f/Mg composites, the holding pressure should be controlled about 15 MPa.

Conclusions

The effect of holding pressure on the performance C_f/Mg composites was studied, and the following conclusions could be drawn:

1. With the increase of the holding pressure from 0.1 to 15 MPa, the porosity in the C_f/Mg composites decreased, the microstructure became compact and the density and the hardness significantly increased. When the holding pressure was in the range of 25–45 MPa, the effect of the holding pressure on the densification was slight.
2. The effect of the holding pressure on the performance of C_f/Mg composites was complicated. The feeding of magnesium alloy was insufficient when the holding pressure was 0.1 or 5 MPa, which led to a poor bonding between carbon fibres and matrix metal, so the fracture surface showed as loose characteristics. When the holding pressure was 15 or 25 MPa, the fracture surface was an obvious step pattern, and the UTS of the C_f/Mg composites could reach 360 MPa. When the holding pressure was 35 or 45 MPa, the carbon fibre was damaged, and the UTS exerted a

downward trend. In order to obtain dense and well mechanical prosperities C_f/Mg composites, the holding pressure should be about 15 MPa in the fabrication of C_f/Mg composites.

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